

## IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of: Kansai Paint Co., LTD.

Serial No.: 10/588,796

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Examiner: PAUL JESSICA M

For: PHOTOCURABLE RESIN COMPOSITION FOR FORMING OPTICAL WAVEGUIDE,

PHOTOCURABLE DRY FILM FOR FORMING OPTICAL WAVEGUDE, AND OPTICAL

WAVEGUIDE

# <u>DECLARA</u>TION

Honorable Commissioner of Patents and Trademarks Washington, D.C. 20231

### Sir :

- I, Genji Imai, hereby declare that:
- I am one of the inventors of the instant invention and
   I am fully familiar with the subject matter thereof;
- 2) I graduated from Toyohashi University of Technology, Faculty of Engineering, Department of Materials Science in March 1987. I obtained a master's degree in Materials Science, from the Graduate School of Toyohashi University of Technology in March 1989. In April 1989, I joined Kansai Paint Co., LTD., assignee of the above-identified application, where I was engaged in research on Photoresists from April 1990 to March 2008. Since April 2008, I have been engaged in research on UV-Coatings as an engineer in CM Laboratory of Kansai Paint Co., LTD.
- 3) the experiments given below were carried out under my general direction and supervision.

#### EXPERIMENTAL DATA

## I. Experiments

- (1)-1. Summary and Purpose of Experiment 1

  Optical waveguides were assembled using a photocurable resin composition or photocurable dry film with a different composition, and the core shape precision of each optical waveguide was evaluated.
- (1)-2. Experimental methods
- (1)-2-1. Production of photocurable resin composition (1)

  Methyl methacrylate (40 g), styrene (20 g), butyl acrylate (20 g)

  and acrylic acid (20 g) were subjected to a radical reaction at 110°C

  to obtain an acrylic resin (resin acid value: 155 mg KOH/g).

  Glycidyl methacrylate (24 g), hydroquinone (0.12 g) and

  tetraethylammonium bromide (0.6 g) were then added to the resin,

  and a reaction was carried out at 110°C for 5 hours while introducing

  air to thereby obtain a photocurable resin. Subsequently, 124 g

  (solids) of the photocurable resin, an aminoalkylphenone

  polymerization initiator (3 g; a product of Ciba Speciality

  Chemicals; trade name "Irgacure 907"), Epicoat EP-828EL (20 g; a

  trade name of Japan Epoxy Resin Co., Ltd.) and ethyl acetate (400 g) were mixed together to obtain photocurable resin composition (1).
- (1)-2-2. Production of photocurable resin composition (2)

  Methyl methacrylate (20 g), styrene (40 g), butyl acrylate (20 g)

  and acrylic acid (20 g) were subjected to a radical reaction at 110°C

to obtain an acrylic resin (resin acid value: 155 mg KOH/g). Glycidyl methacrylate (24 g), hydroquinone (0.12 g) and tetraethylammonium bromide (0.6 g) were then added to the resin, and a reaction was carried out at 110°C for 5 hours while introducing air to thereby obtain a photocurable resin. Subsequently, 124 g (solids) of the photocurable resin, an aminoalkylphenone polymerization initiator (3 g; a product of Ciba Speciality Chemicals; trade name "Irgacure 907"), Epicoat EP-828EL (20 g; a trade name of Japan Epoxy Resin Co., Ltd.) and ethyl acetate (400 g) were mixed together to thereby obtain photocurable resin composition (2).

(1)-2-3. Production of photocurable resin composition (3)

Methyl methacrylate (40 g), butyl acrylate (40 g) and acrylic acid

(20 g) were subjected to a radical reaction at 110°C to obtain an acrylic resin (resin acid value: 155 mg KOH/g). Glycidyl methacrylate (24 g), hydroquinone (0.12 g) and tetraethylammonium bromide (0.6 g) were then added to the resin, and a reaction was carried out at 110 °C for 5 hours while introducing air, to thereby obtain a photocurable resin. Subsequently, 124 g (solids) of the photocurable resin, an aminoalkylphenone polymerization initiator (3 g; a product of Ciba Speciality Chemicals; trade name "Irgacure 907"), Hydrogenated bisphenol A diglycidyl ether (20 g) and ethyl acetate (400 g) were mixed together to obtain photocurable resin composition (3).

(1)-2-4. Production of photocurable resin composition (4)

124 g (solids) of the same photocurable resin as obtained in (1)-2-3,
an aminoalkylphenone polymerization initiator (3 g; a product of
Ciba Speciality Chemicals; tradename "Irgacure 907"), Epicoat

EP-828EL (20 g; a tradename of Japan Epoxy Resin Co., Ltd.) and ethyl
acetate (400 g) were mixed together, to thereby obtain photocurable
resin composition (4).

### (1)-2-5. Production of photocurable dry film (D-1)

Photocurable resin composition (1) was applied on a polyethylene terephthalate film (film thickness:  $25~\mu m$ ) with a knife edge coater, and dried at  $80^{\circ}C$  for 30 minutes to obtain photocurable dry film (D-1).

### (1)-2-6. Production of photocurable dry film (D-2)

Photocurable resin composition (2) was applied on a polyethylene terephthalate film (film thickness:  $25 \, \mu m$ ) with a knife edge coater, and dried at  $80\,^{\circ}$ C for 30 minutes to obtain photocurable dry film (D-2).

# (1)-2-7. Production of photocurable dry film (D-3)

Photocurable resin composition (3) was applied on a polyethylene terephthalate film (film thickness:  $25~\mu m$ ) with a knife edge coater, and dried at  $80^{\circ}$ C for 30 minutes to obtain photocurable dry film (D-3).

# (1)-2-8. Production of photocurable dry film (D-4)

Photocurable resin composition (4) was applied on a polyethylene terephthalate film (film thickness:  $25 \, \mu m$ ) with a knife edge coater, and dried at  $80\,^{\circ}$ C for 30 minutes to obtain photocurable dry film (D-4).

### (1)-2-9. Production of optical waveguide(1)

# [1] Formation of lower cladding layer

Photocurable resin composition (1) was applied on the surface of a silicon substrate by spin coating, and irradiated for 100 seconds with ultraviolet rays having a wavelength of 365 nm and an illuminance of 10 mW/cm² to form a lower cladding layer with a thickness of 40  $\mu$ m. The lower cladding layer was then cured by heating at 150 °C for 60 minutes.

#### [2] Formation of the core

Photocurable resin composition (2) was applied on the lower cladding layer by spin coating, and dried at 80 °C for 30 minutes. The applied composition was cured by irradiation for 100 seconds with ultraviolet rays having a wavelength of 365 nm and an illuminance of 10 mW/cm² via a photomask having a pattern consisting of 30  $\mu$ m-wide lines. The substrate with the ultraviolet ray-irradiated resin composition layer was immersed in a developer made of an aqueous 1.8 wt.% tetramethylammonium hydroxide solution to dissolve the unexposed portion of the resin composition layer and then tried. A core having a pattern consisting of 30  $\mu$ m-wide lines was thus formed.

The core was then cured by heating at 150 °C for 60 minutes.

## [3] Formation of upper cladding layer

Photocurable resin composition (1) was applied on the top surfaces of the core and lower cladding layer by spin coating, and irradiated for 100 seconds with ultraviolet rays having a wavelength of 365 nm and an illuminance of 10 mW/cm² to form an upper cladding layer with a thickness of 40  $\mu$ m. The upper cladding layer was then cured by heating at 150 °C for 60 minutes.

The obtained optical waveguide was evaluated according to the above criteria. As a result, the transmission loss was rated as A; the core gap as A; the core shape precision as A; the core-covering properties as B; and workability as B.

The relative refractive index difference between the core and cladding layers was at least 0.1%.

### (1)-2-10. Production of optical waveguide (2)

### [1] Formation of lower cladding layer

Photocurable dry film (D-1) was transferred onto a surface of a silicon substrate by atmospheric-pressure hot roll bonding (temperature: 100 °C), and cured by irradiation for 100 minutes with ultraviolet rays having a wavelength of 365 nm and an illuminance of 10 mW/cm<sup>2</sup>. The polyethylene terephthalate film was then peeled off to form a lower cladding layer with a thickness of 40 µm. The

lower cladding layer was then cured by heating at 150  $^{\circ}\text{C}$  for 60 minutes.

### [2] Formation of core

Subsequently, photocurable dry film (D-2) was transferred onto the lower cladding layer by atmospheric-pressure hot roll bonding (temperature:  $100\,^{\circ}$ C). The transferred dry film was cured by irradiation for 100 seconds with ultraviolet rays having a wavelength of 365 nm and an illuminance of 10 mW/cm² via a photomask having a pattern consisting of 30 µm-wide lines. The polyethylene terephthalate film was then peeled off. Thereafter, the substrate with the ultraviolet ray-irradiated resin composition layer was immersed in a developer made of an aqueous 1.8 wt.% tetramethylammonium hydroxide solution to dissolve the unexposed portion of the resin composition layer, followed by drying. A core having a pattern consisting of 30 µm-wide lines was thus formed. The core was then cured by heating at 150 °C for 60 minutes.

#### [3] Formation of upper cladding layer

Photocurable dry film (D-1) was transferred onto the top surfaces of the core and lower cladding layer by atmospheric-pressure hot roll bonding (temperature: 100°C), and cured by irradiation for 100 seconds with ultraviolet rays having a wavelength of 365 nm and an illuminance of 10 mW/cm<sup>2</sup>. The polyethylene terephthalate film was then peeled off to form an upper cladding layer with a thickness of 40 µm. Thereafter, the upper cladding layer was cured by heating

at 150 °C for 60 minutes.

The obtained optical waveguide was evaluated according to the above criteria. As a result, the transmission loss was rated as A; the core gap as A; the core shape precision as A; the core-covering properties as A; and workability as A.

The relative refractive index difference between the core and cladding layers was at least 0.1%.

# (1)-2-11. Production of optical waveguide (3)

The procedure of (1)-2-9 was followed except for the use of photocurable resin compositions (3) and (4) in place of photocurable resin compositions (1) and (2), respectively, to thereby obtain optical waveguide (3).

# (1) -2-12. Production of optical waveguide (4)

The procedure of (1)-2-10 was followed except for the use of photocurable dry films (D-3) and (D-4) in place of photocurable dry films (D-1) and (D-2), respectively, to thereby obtain optical waveguide (4).

#### Evaluation of core shape precision

The obtained optical waveguides were evaluated visually with respect to core shape precision.

### (1) -2. Result

Table 1 shows core shape precision of each sample.

Table 1

	Core shape precision
Optical waveguide (1)	А
Optical waveguide (2)	A
Optical waveguide (3)	В
Optical waveguide (4)	В

"A" indicates that the core was not deformed by the upper cladding layer; and "B" indicates that the core was deformed by the upper cladding layer.

As is clear from Table 1, in a photocurable resin composition comprising:

- (A) a carboxy-containing unsaturated <u>acrylic</u> resin obtained by reacting a carboxy-containing acrylic resin (a) with an epoxy-containing unsaturated compound (b);
- (B) a solvent; and
- (C) a <u>bisphenol-type</u> epoxy resin or a novolac-type epoxy resin, when the carboxy-containing acrylic resin (a) is made of a material containing styrene (optical waveguides (1) (2)), the optical waveguide obtained using the photocurable resin composition achieves an remarkable effect, i.e., excellent core precision.

In contrast, when the carboxy-containing acrylic resin (a) is made

of a material containing no styrene (optical waveguides (3) (4)), the core precision is significantly lowered.

I, the undersigned, declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Gonjhos

Date: June 3, 2009